



Detached Solidification of Germanium-Silicon Crystals on the ISS



M. P. Volz^a, K. Mazuruk^b, A. Cröll^{b,c}

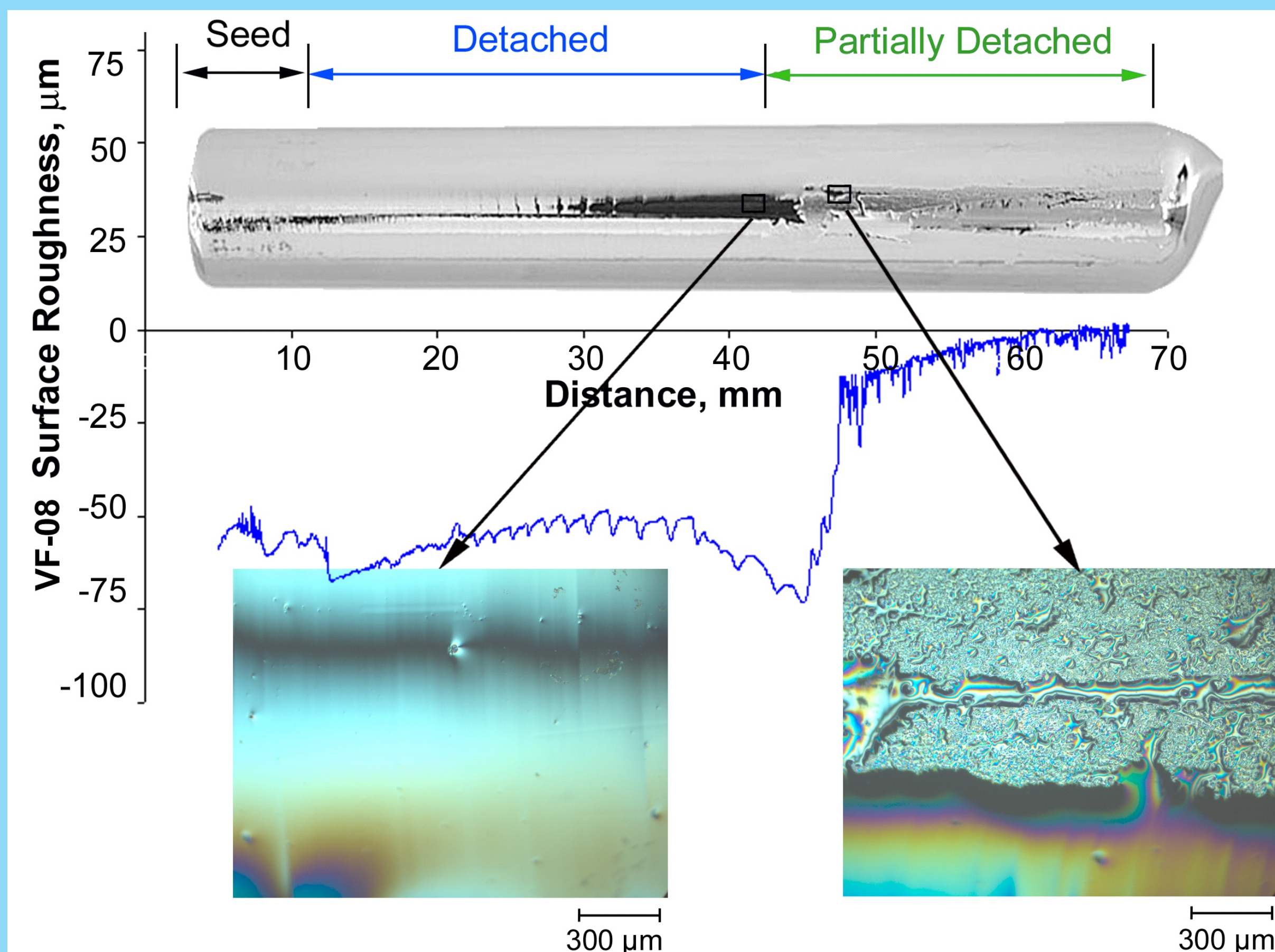
^aNASA Marshall Space Flight Center, EM31, Huntsville, AL 35812, USA

^bUniversity of Alabama in Huntsville, Huntsville, AL 35762, USA

^cKristallographie, University of Freiburg, Freiburg, Germany

Abstract

A series of $\text{Ge}_{1-x}\text{Si}_x$ crystal growth experiments are planned to be conducted in the Low Gradient Furnace (LGF) onboard the International Space Station. The primary objective of the research is to determine the influence of containment on the processing-induced defects and impurity incorporation in germanium-silicon alloy crystals. A comparison will be made between crystals grown by the normal and “detached” Bridgman methods and the ground-based float zone technique. Crystals grown without being in contact with a container have superior quality to otherwise similar crystals grown in direct contact with a container, especially with respect to impurity incorporation, formation of dislocations, and residual stress in crystals. “Detached” or “dewetted” Bridgman growth is similar to regular Bridgman growth in that most of the melt is in contact with the crucible wall, but the crystal is separated from the wall by a small gap, typically of the order of 10-100 microns. Long duration reduced gravity is essential to test the proposed theory of detached growth. Detached growth requires the establishment of a meniscus between the crystal and the ampoule wall. The existence of this meniscus depends on the ratio of the strength of gravity to capillary forces. On Earth, this ratio is large and stable detached growth can only be obtained over limited conditions. Crystals grown detached on the ground exhibited superior structural quality as evidenced by measurements of etch pit density, synchrotron white beam X-ray topography and double axis X-ray diffraction.



Detached growth of germanium

The crystal shown above was grown in a pyrolytic boron nitride (pBN) ampoule. The high wetting angle of molten Ge on pBN leads to detachment. The surface profilometer indicates a gap width of about 60 microns during the detached part of growth. The surface of the detached crystal is quite smooth. In contrast, the surface of the attached portion of growth has the roughness of the interior surface of the ampoule.

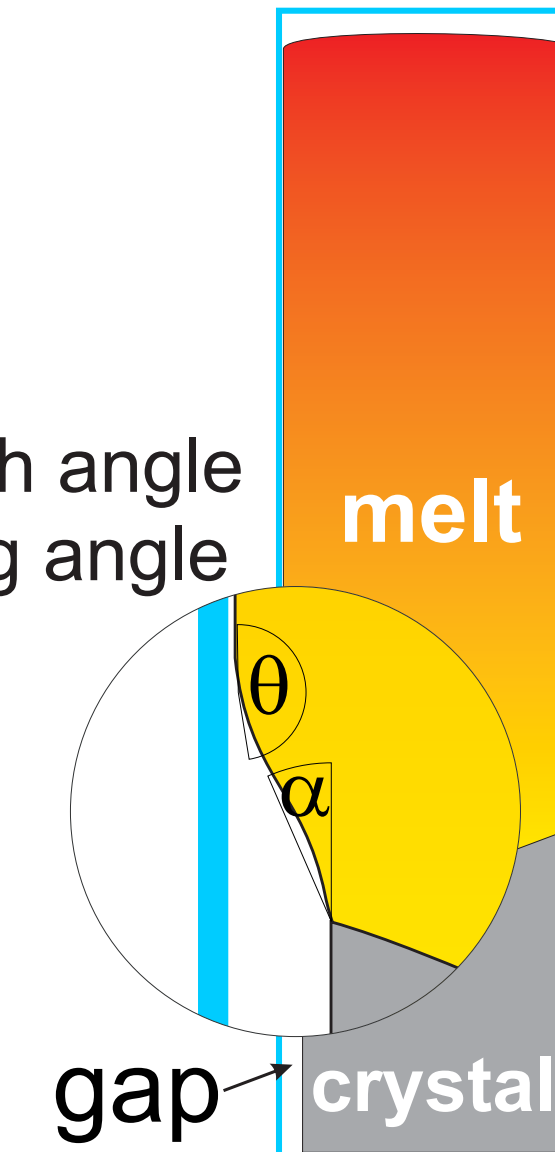
Float-Zone growth



Bridgman growth



Detached Bridgman

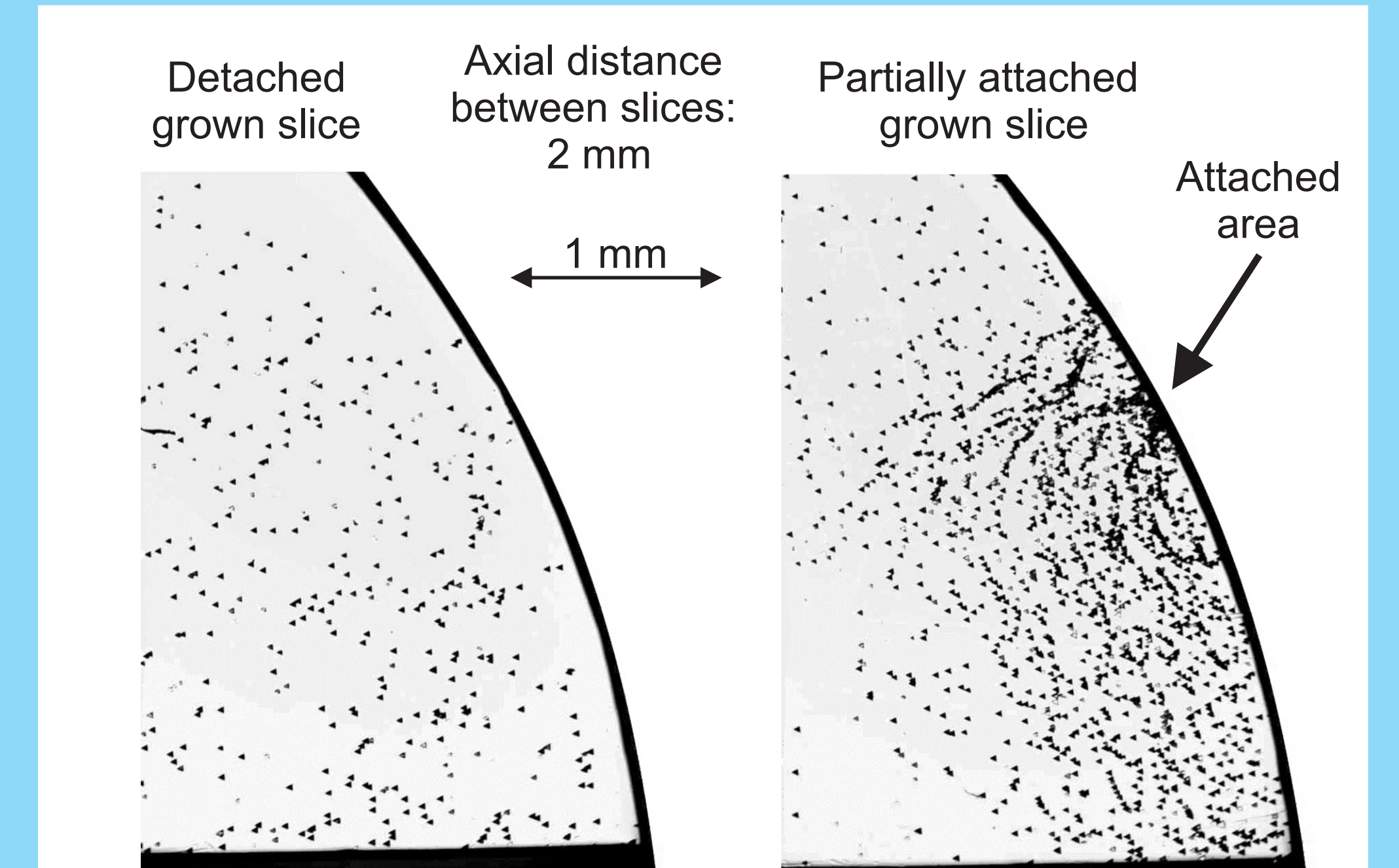
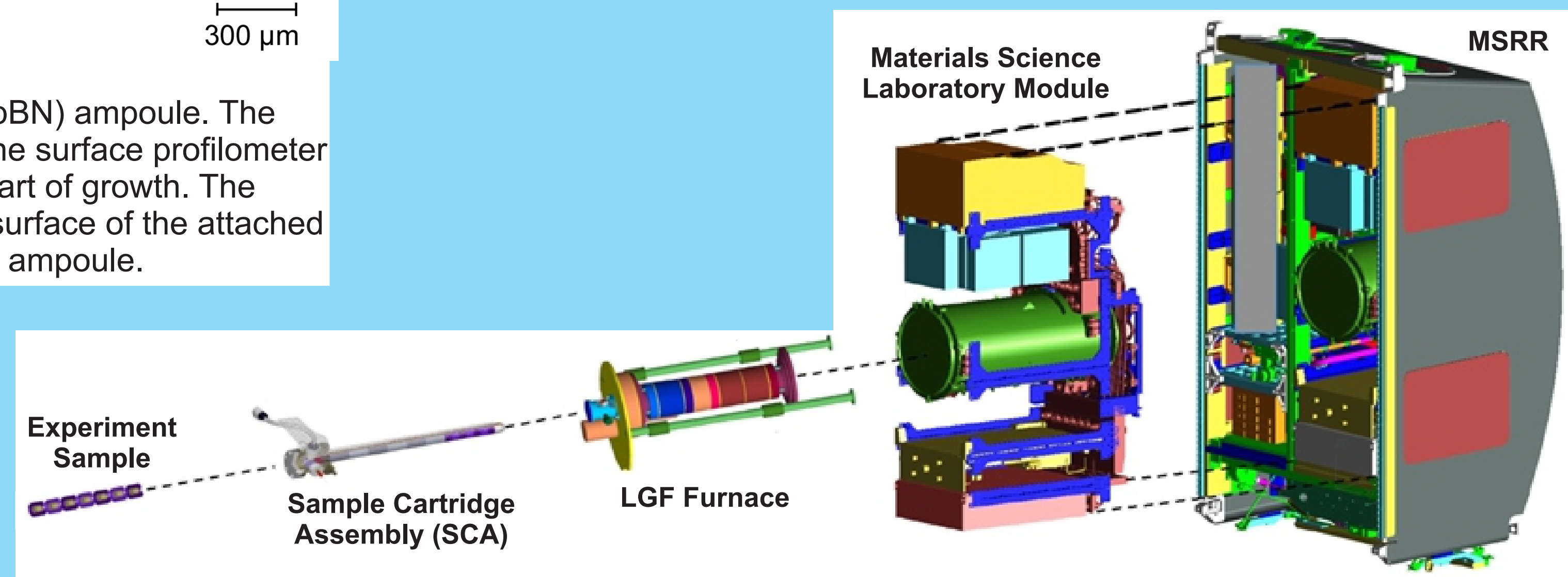


Crystal Growth Methods

In Float-Zone processing, the melt does not make contact with a container wall. However, on Earth, the maximum diameter is typically limited to about 10 mm. In both the normal and detached Bridgman processes, the melt is in contact with the ampoule wall. The ampoule is translated with respect to a thermal gradient and the melt directionally solidifies. However, in the detached Bridgman process, a meniscus forms at the bottom of the melt between the crystal and ampoule wall. The size of the gap below this meniscus is typically of the order of 10-100 microns.

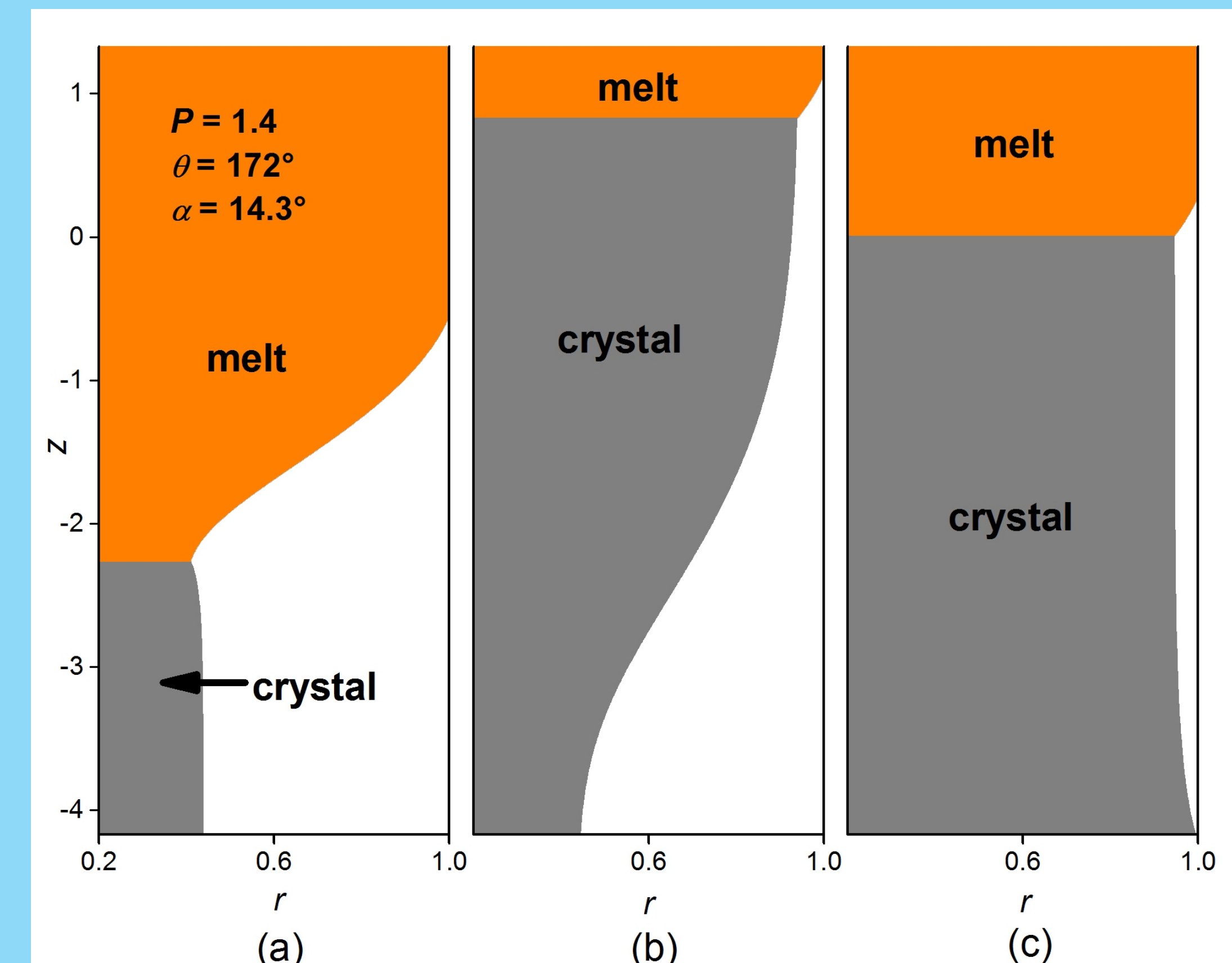
Flight Experiments

- A series of 10 $\text{Ge}_{1-x}\text{Si}_x$ samples will be processed in the Low Gradient Furnace (LGF) in the Materials Science Research Rack (MSRR) on the ISS.
- The samples are currently scheduled to be launched to the ISS in 2017.
- The experiments will vary parameters that are key to a better understanding of the theory of detached growth: pressure differential across the meniscus, contact angle, growth angle, and Bond number (ratio of capillary and gravitational forces).
- The samples will be compared to identically processed samples on Earth.



Etch-pit density measurements

D-shaped (111)-oriented radial wafers were cut perpendicular to the growth direction. They were polished and then etched with the Billig etchant (12g KOH and 8g $\text{K}_3[\text{Fe}(\text{CN})_6]$ dissolved in 100 ml H_2O at approximately 85 C). The localized etch pit density increases where the crystal attaches partially to the wall.



Evolution of crystal shapes in microgravity

Examples of crystal and meniscus shapes in microgravity. The crystal evolution will depend on the pressure differential across the meniscus and the contact and growth angles. (a) inward crystal growth towards eventual meniscus collapse; (b) outward growth towards stable growth with a constant radius; (c) inward growth towards stable growth with a constant radius.